

Novel Silica-Based Ion Exchange Resin

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INTRODUCTION

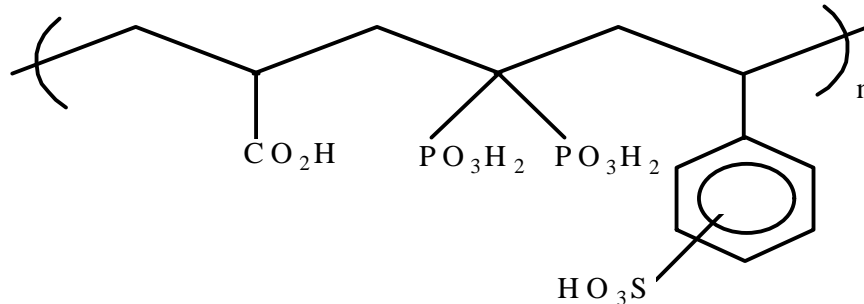
Conventional sulfonic acid and amine type ion exchange resins are widely used in many commercial applications including the softening or deionizing of water, treatment of industrial wastewater and the production and recovery of metals. However, these resins have limited use in specialized applications such as the preconcentration and removal of trace metals for the purpose of analysis, the treatment of industrial wastewaters to the parts per billion level, and the removal of radionuclides from highly acidic waste streams. Specialized applications require more selective ion exchange resins that target one or more metal ions so their capacity is not exhausted removing the more common cations (sodium, magnesium, calcium) or anions (chlorides, sulfates, nitrates). In response to the need for greater selectivity, a class of resins called chelating ion exchange resins was developed and commercialized. Chelating resins provide a dramatic improvement in ion selectivity relative to traditional ion exchange resins but still suffer from two distinct drawbacks: slow metal uptake kinetics and a limited operating pH range. Slow reaction kinetics result in the underutilization of resin capacity and premature ion breakthrough. These, in turn, lead to higher capital and operating costs for the systems in which the chelating resins are used. The inability of chelating resins to remove target metal ions selectively from highly acidic solutions also prevents their effective use with waste streams such as acidic nuclear waste, acid mine drainage, and metal processing streams.

The shortcomings of chelating resins have been addressed by the development of a new class of ion exchange resins called dual mechanism bifunctional polymers (DMBPs). Like conventional ion exchange resins, DMBPs use hydrophilic cation exchange ligands that have rapid uptake kinetics; and like chelating resins, DMBPs use chelating ligands to achieve selectivity for one or more metals. By combining hydrophilicity and selectivity, DMBPs overcome the limitations of conventional resins (selectivity) and of chelating resins (uptake kinetics). The result is a resin that quickly recognizes and removes targeted metals from waste, remediation and process streams.

Eichrom's Diphonix® resin represents the first DMBP to be widely released as a commercial product. Diphonix resin was developed by researchers at the DOE's Argonne National Laboratory and at the University of Tennessee. Eichrom licensed the technology and has successfully commercialized it in applications as diverse as metallurgical processing, mixed waste treatment, semiconductor manufacturing, and electric power plant water treatment.

Eichrom's Diphonix resin, illustrated in Figure 1, resembles a conventional ion exchange resin in its use of sulfonic acid ligands on a styrene-divinylbenzene matrix. Therefore, Diphonix resin exhibits the rapid exchange kinetics that allow economical operation of ion exchange systems. Unlike conventional resins, Diphonix resin contains chelating ligands that reject common elements and limit the resin's uptake to targeted metal ions. These chelating ligands are gem-diphosphonic acid groups that recognize and remove the targeted metals and reject the more common elements such as sodium, calcium, and magnesium. Moreover, systems using Diphonix resin look and operate no differently than do systems using conventional ion exchange resins. No specialized equipment or operator training is required and regenerating the resin is similar to the regeneration of other ion exchange resins.

Figure 1



Diphonix resin polymer matrix

Diphonix resin also addresses another limitation of chelating resins: operation at low pH ranges. The gem-diphosphonic acid ligands of the Diphonix resin tend to form protonated metal complexes. As a result, the resin retains its selectivity and capacity for metals under conditions that are typically too acidic for appreciable metal complexation by conventional and chelating ion exchange resins.

Physical and chemical properties of Diphonix resin are shown below:

Diphonix Resin Properties

Ionic Form:	Hydrogen
Density:	Ranges from 1.05 to 1.11 g/mL depending on mesh size.
Moisture Content:	Varies with crosslinkage specified. Typical value is 70% for 5% divinylbenzene cross-linked beads.
Particle Size Ranges:	Available in 20-50 mesh, 50-100 mesh and 100-200 mesh.
Capacity:	Hydrogen capacity = 5-6 mmol/g Phosphorus content \approx 1.4 millimoles P per gram of dry resin

Capacity in the above table refers to the maximum number of phosphorus sites (expressed as millimoles P per gram of dry resin) available for binding metal ions and is called total theoretical capacity.

Currently, Diphonix resin is synthesized by a patented process involving copolymerization of a tetraalkylvinylidene diphosphonate with styrene, divinylbenzene, and acrylonitrile followed by sulfonation with concentrated sulfuric acid. Determining a method for effectively copolymerizing the vinylidene diphosphonate ester was a major achievement because of the steric hindrance imposed on the vinylidene group by the diphosphonate group. This difficulty was overcome by using another relatively small monomer, acrylonitrile, as a carrier to induce polymerization of the vinylidene diphosphonate.

The major technical challenge of synthesizing silica-based Diphonix resin is incorporation of diphosphonic acid groups onto silica particles in quantities that provide metal loading capacity, kinetics, and acid stability comparable to the polystyrene-based Diphonix resin. The synthesis of polystyrene-based Diphonix resin as described above is achieved in a two step process that includes a copolymerization step in which the diphosphonic acid groups are incorporated throughout the organic matrix and a sulfonation step that adds the sulfonic acid group and hydrolyzes the phosphate esters. The synthesis of silica-based Diphonix resin will require that the diphosphonic acid groups be chemically bonded onto the surfaces of the silica particles in a multi-step process. The silica particles are highly porous and include both micro and macro pores in a rigid solid matrix of silicon dioxide that contains little or no moisture. The silica matrix is functionalized by attaching diphosphonic acid groups onto the silanol sites contained on the macroporous surface.

Silanol sites in the silica micropores are not functionalized and are rendered inaccessible by the synthesis process.

Studies by Eichrom indicate that diphosphonic acid groups can be chemically incorporated onto silica by chemically bonding a carbon-containing “anchor” onto the silanol sites of the silica support, grafting a polymeric chain onto the “anchor”, then functionalizing the chain with diphosphonic esters that are subsequently hydrolyzed to diphosphonic acid groups. Because the method of introduction for the gem-diphosphonic acid groups is different for the proposed resin than it is for the polystyrene-based Diphonix resin, Eichrom expects to have more control over the silica-based resin’s phosphorus content. The phosphorus content is a direct measure of the chelating binding sites on the resin and therefore of the resin’s capacity. Eichrom expects to be able to tailor the capacity of the proposed resin for binding metals by controlling the length of the polymer chain containing the diphosphonate acid ligands. Increasing the length of the polymeric chain increases the number of diphosphonic acid ligands and vice versa.

Because of silica’s high porosity, the metal uptake kinetics are expected to be comparable to those of the polystyrene-based Diphonix resin. However, sulfonation of the silica-based resin might be required to provide the sulfonic acid groups necessary to improve the kinetics by increasing the hydrophilicity of the resin. The sulfonation process of the silica-based resin would be similar to the current sulfonation process of the polystyrene-based Diphonix resin.

OBJECTIVES

The objective of this work is to synthesize commercial quantities of a silica-based ion exchange resin with the same or better metal ion selectivity, metal uptake kinetics and acid stability as the commercially available polystyrene-based Diphonix ion exchange resin. Diphonix resin contains gem-diphosphonic acid groups that function as chelating ligands and that exhibit significant selectivity for absorbing actinides and certain other classes of metals over the common elements sodium, calcium, and magnesium. Diphonix resin retains its selectivity and metal uptake capacity even under highly acidic conditions because of the unique metal ion coordination ability of the diphosphonic acid groups.

APPROACH

The technical approach of this proposed project is to synthesize several silica-supported diphosphonic acid resins, to evaluate different commercially-available silica supports, and to optimize the chemical grafting and polymerization techniques. The laboratory-scale synthesis studies will be confirmed in pilot facilities that will permit development of manufacturing economics and engineering data related to production scale-up. The synthesized resins will be characterized for reaction kinetics, resin capacity, particle size, and particle porosity. Those resins meeting predetermined success criteria for each characteristic will then undergo further laboratory tests including actinide selectivity and uptake kinetics, effects of competing cations and complexing anions, and determination of

radiolytic stability. Finally, stripping conditions will be determined to allow recovery and recycling of certain metals from their respective waste streams. Vittrification studies are also scheduled to determine the advantage of a silica-based resin over a hydrocarbon-based resin for this long term disposal option.

RESULTS

Preliminary laboratory studies indicate that a silica-based resin with diphosphonic acid functional groups can be synthesized. However, extensive work remains to optimize the process. Eichrom has developed a laboratory-scale resin synthesis strategy based on these studies that will include varying three critical parameters: silica porosity, reaction time, and reactant/initiator concentrations. The objective of the additional laboratory studies is to optimize the resin formulation. The success of these preliminary investigations mitigates the risk of failing to produce a silica-based resin with the same diphosphonic acid functionality as polystyrene-based Diphonix resin.

Comprehensive studies have been completed at Eichrom and at Argonne National Laboratory to characterize the performance of Diphonix resin over a broad range of nitric and hydrochloric acid conditions and inorganic salt loadings. The resin characteristics quantified were metal uptake values, kinetic behavior, and metal loading and stripping capacities. The results of the studies are discussed below. Included with this discussion of the performance characteristics of Diphonix resin is a discussion of the proposed method of incorporating similar characteristics into a silica-based resin.

Batch Uptake Studies. Batch uptake studies measure the quantity of metal ions absorbed from a solution onto the resin after sufficient contact to reach equilibrium. Data from the batch uptake tests are used to calculate distribution ratios or D values (see equation (1)). D values represent a resin's ability to remove a given metal ion from a solution of defined pH and ionic concentration. The higher the distribution ratio, the more targeted metal ions are taken up by the resin's functional groups and the better the decontamination factor that should be achievable with the resin. In other words, a high D value indicates the resin possesses the ability to remove more completely a targeted metal ion from solution, yielding an effluent stream with greatly reduced concentration of the target species.

$$(1) \quad D = ((A_o - A_f)/W)/(A_f/V) \quad \text{where:} \quad \begin{array}{l} A_o = \text{aqueous phase activity before} \\ \quad \text{equilibration} \\ A_f = \text{aqueous phase activity after} \\ \quad \text{equilibration} \\ W = \text{weight of dry resin, grams} \\ V = \text{aqueous phase volume, mL} \end{array}$$

In studies conducted at Argonne National Laboratory, the uptake of several actinide ions (U(VI), Pu(IV), Th(IV), and Am(III)) from nitric and hydrochloric acid solutions was determined.¹ Figure 2 shows the nitric acid dependencies of the D values, of U(VI),

Pu(IV), Th(IV), and Am(III). The most striking feature of the data in Figure 2a is the high retention of hexavalent uranium (D values from 10^6 to 10^4) and tetravalent actinides (D values from 10^7 to 10^4) by Diphonix resin in the entire acidity range from 0.01M to 10M nitric acid. The retention of the trivalent americium is more typical of sulfonic acid type resins with D values declining from 10^6 (0.1M nitric acid) to 10^2 (10M nitric acid). Similar behavior was observed with analogous studies performed in a hydrochloric acid matrix. Additional studies were conducted to determine the uptake of some trivalent transition and post transition metals.² Figure 2b shows the distribution values of the trivalent ions, iron, europium, aluminum, chromium and bismuth. The most significant feature is the behavior of Fe(III) which strongly resembles the curves reported for Pu(IV) and Np(IV).

Figure 2

2a

2b

(SEE END OF TEXT)

Nitric Acid Dependencies of Actinide Ion Uptake by Diphonix Resin

Nitric Acid Dependencies of Trivalent Ion Uptake by Diphonix Resin

Because the same gem-diphosphonic acid group will be incorporated into the proposed silica-based resin, we expect to achieve metal uptake on a silica-based Diphonix resin comparable to the polystyrene-based resin.

Kinetic Studies. As mentioned above, it is known that some chelating resins (carboxylic, iminodiacetic, and phosphonate) exhibit metal uptake kinetics that are much slower than traditional sulfonic and quaternary ammonium type resins. In general, this is the principal flaw of chelating ion exchange resins and limits their applicability despite high selectivity.

The low hydrophilicity of most chelating resins makes it difficult for metal ions to migrate to the binding sites of the chelating resin. As described above, DMBPs offer an opportunity to combine the selectivity advantages of a chelating resin with the rapid

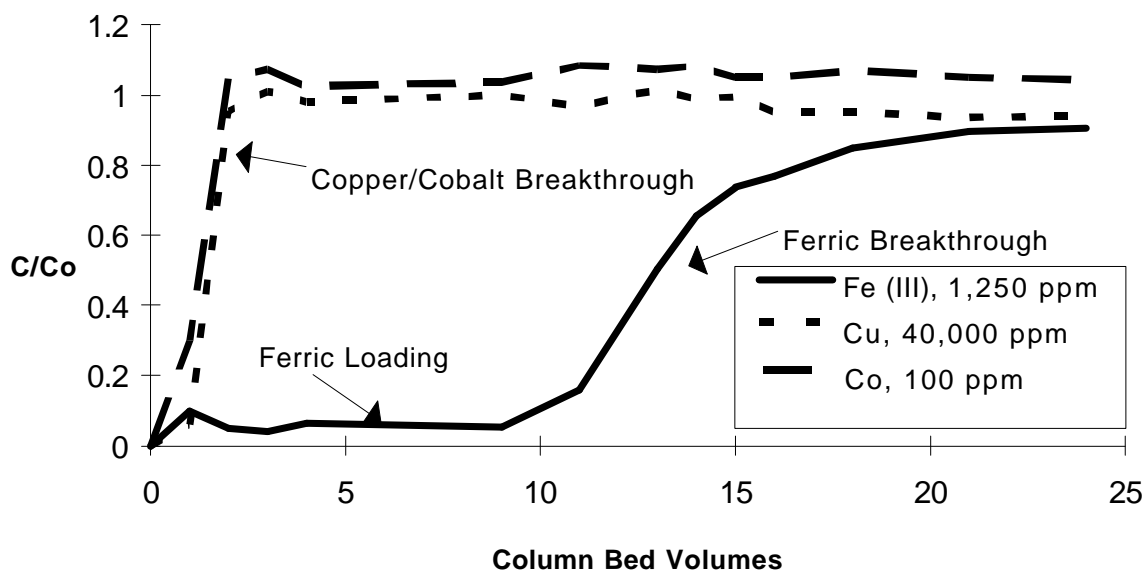
kinetics of a conventional resin. Diphonix resin is a good example of this benefit, where the selectivity of the diphosphonic acid ligands was augmented by the incorporation of hydrophilic sulfonic acid groups. A series of experiments was conducted and established the paramount importance of the sulfuric acid groups in obtaining practically useful rates of metal ion uptake.³

In the proposed silica-based resin syntheses, silica gels of varying porosity will be studied. Due to the high level of porosity of silica and the related high accessibility of the diphosphonate binding sites, we expect to find metal uptake kinetics comparable to the sulfonated polystyrene-based Diphonix resin. Sulfonation of silica-based resin is also technically feasible and will be explored if enhanced kinetics are required.

Column Loading Studies. The total theoretical capacity of Diphonix resin for a specific metal ion depends on the molecular weight and valence of the ion. For example, the total capacity of Diphonix resin for Fe(III) is 30 mg per gram of resin, whereas for U(VI) it is 166 mg per gram of resin. In practice, the operating capacity of a column is the ion exchange capacity realized in a given application under specific conditions. The column operating capacity is a complex value and varies depending on solution parameters such as concentration of the targeted ions, the resin selectivity for the targeted ions, the flowrate, temperature, and the presence or absence of competing ions. The column operating capacity is expressed as the point (in terms of resin bed volumes) at which the targeted ions are observed in the column effluent, known as “breakthrough”. Operating capacity can also be expressed as mg’s of targeted ion uptake per gram of resin and is usually a fraction of the total theoretical capacity.

Recent loading studies at Eichrom were carried out on strongly acidic (150 grams per liter sulfuric) copper electrolyte solutions and illustrate the selectivity of the resin for Fe(III) over the divalent metal species, cobalt and copper. Figure 3 shows a rapid breakthrough of divalent species. Fe(III) continues to load on the Diphonix resin column until approximately 10 column volumes have passed through the column. The similarity of the distribution value curves (Figures 2a and 2b) for Fe(III) and Pu(IV) and Th(IV) indicate that tetravalent actinides can be expected to behave like Fe(III) under strongly acidic loading conditions. Therefore, Fe(III) is a good predictor of tetravalent actinide loading.

Figure 3



Diphonix Resin Breakthrough Curves for Copper Electrolyte Solution
 C_o = Influent Metal Concentration
 C = Effluent Metal Concentration

In addition, pilot plant studies using columns of Diphonix resin were also conducted at Argonne National Laboratory for removal of U(VI) from a mixed laboratory waste stream.⁴ The results showed that uranium was reduced 300,000 fold to 0.2 ppb. The uranium was subsequently successfully stripped from the resin with 1-hydroxyethane-1,1-diphosphonic acid (HEDPA) and recovered (see discussion in Stripping Studies).

The project work plan includes column loading studies that will be performed with non-radioactive and radioactive solutions representative of DOE waste streams. Because the silica-based Diphonix resin will contain the same diphosphonic acid groups as the polystyrene-based resin, it is expected to yield analogous metal loading results.

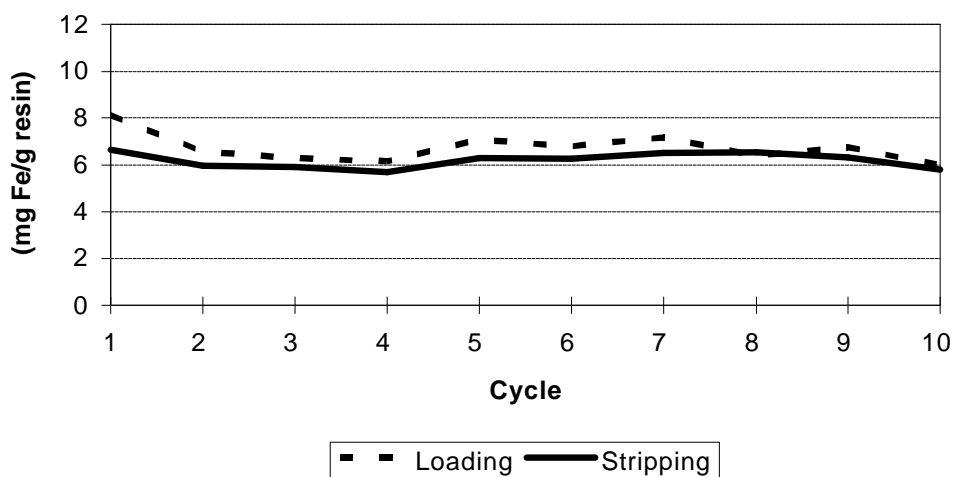
Stripping Studies. A variety of stripping methods has been evaluated for recovering metals from Diphonix resin and allowing reuse of the resin. Stripping uranium and the tetravalent actinides from Diphonix resin generally requires the use of a soluble diphosphonate extractant. In the Argonne National Laboratory study that demonstrated decontamination of Davies-Gray waste (highly acidic uranium processing waste), decontamination factors of 10^5 to 10^7 were observed using Diphonix resin; the uranium was recovered essentially quantitatively from the Diphonix resin columns by stripping with a solution of hydroxyethane-1,1-diphosphonic acid (HEDPA).⁴ Even though the stripping solution now contained uranium, the net effect was to reduce the original waste volumes more than 100 fold. Horwitz et al. also suggests that uranium can be removed effectively from Diphonix resin as a basic carbonato complex with potassium carbonate as the

stripping reagent.¹ While it has been demonstrated that actinides and other metals can be removed effectively from the resin, it may often be preferable to leave actinides on the Diphonix resin or the silica-based analog proposed herein. We are proposing the silica-based analog of Diphonix resin partially because it may represent a more readily vitrifiable solid waste form.

Trivalent americium and most transition metals can be stripped from Diphonix resin with varying concentrations of mineral acids (2-6 M).² In the copper electrolyte study referred to above, a novel reductive strip of iron was performed. Fe(III) is complexed quite tightly by Diphonix resin but reduction of Fe(III) on the column to Fe(II) with SO₂ allowed an effective regeneration of resin capacity. A series of load/strip cycles, illustrated in Figure 4, demonstrated that bead performance was unchanged over 10 cycles. The operating capacity of the Diphonix resin in this application is 6-8 mg iron per gram of resin. Stripping efficiency of 70-80 percent per cycle afforded an economical balance of loading and stripping cycles without an effect on the operating capacity.

It is our expectation that the proposed silica-based Diphonix resin will exhibit similar loading and stripping performance relative to the gel-type resin.

Figure 4



Iron Loading and Stripping Cycles for Copper Electrolyte Solution

Technical Risks. The technical risks associated with the performance of the silica-based resin are mitigated by the enormous quantity of data developed at Argonne National Laboratory and at Eichrom characterizing the performance of polystyrene-based Diphonix resin.¹⁻⁵ Eichrom has developed a strategy for evaluating the performance of a silica-based resin based on this previous work that is efficient and limits the risk of resin performance failure. The technical and financial risks inherent in the synthesis of a new material are also mitigated by the go/no go decision that occurs after preliminary resin

performance tests such as metal uptake and kinetic tests. Favorable results will justify continuing with dynamic column loading and stripping studies and vitrification experiments.

APPLICATION

The new, silica-based resin functionalized with diphosphonic acid ligands can be used in a number of environmental restoration and waste management situations involving the processing of low-level, transuranic (TRU) and high-level radioactive wastes. It can also be used for processing liquid mixed waste (waste that contains low level radioactivity and hazardous constituents) including mixed wastes contaminated with organic compounds. The advantages expected of the new resin technology are:

- increased radiolytic stability of the silica-based resin relative to its polystyrene-based counterpart (Diphonix resin)
- a resultant waste form that is amenable to disposal without additional treatment or that can be vitrified more readily than the conventional ion exchange resins
- better silica matrix stability at highly basic pH (pH above 11) than other silica ion exchangers
- performance improvements with waste streams containing organic compounds (> 1%)

Radiolytic Stability A pressing challenge posed by wastes of the DOE complex is radioactivity and the concomitant need for radiolytically stable treatment technologies. Radiation damage to polystyrene-based ion exchange resins is caused by the rupture of the carbon-carbon bonds in the resin's, crosslinkages leading to resin degradation and loss of metal uptake capacity. Generally, cation exchangers have higher radiolytic stability than anion exchangers and highly crosslinked exchangers are more stable than those with a low degree of crosslinkage.⁶ A silica-based support, in contrast, consists of silicon and oxygen bonded together in a rigid, highly porous matrix. The silicon-oxygen bonds are much stronger than the carbon-carbon bonds and are much less susceptible to radiation degradation. Therefore, a silica support is expected to be more stable in radioactive conditions.

Vitrification The DOE is considering vitrification for treatment of its low-level and mixed waste because of the potential to lower disposal costs and to minimize emissions from the vitrified product or the final waste form. The vitrification process has been proven to work on high-level waste and is currently declared Best Demonstrated Available Technology (BDAT) for disposing of high-level radioactive waste.⁷

Vitrification is a thermal process that converts hazardous and radioactive contaminants into a glass or glass-like substance. In the vitrification process, the inorganic contaminants are melted or dissolved into molten liquid that when cooled forms a glass product that chemically incorporates or encapsulates the contaminants. Vitrification also destroys organic contaminants. The composition of the waste stream to be vitrified is important. The waste must contain a favorable proportion of glass forming compounds (silicon, aluminum and iron oxides) or these materials must be added to obtain the appropriate level. Since a silica-based Diphonix resin will be essentially 100% silicon dioxide compared a polystyrene-based exchanger which would contain no silicon dioxide material, less glass-forming material will be required for vitrification. Therefore, a silica-based Diphonix ion exchange resin should be more readily vitrifiable than polystyrene resins, and, in fact, could itself be used as a glass-forming additive.

pH Characteristics It is known that silica-based material will normally dissolve under highly basic conditions (pH above 11). However, preliminary laboratory studies have shown that silica-based Diphonix resin is not subject to dissolution in highly basic conditions and therefore maintains its metal uptake capacity. The reason for matrix stability under highly basic conditions is due to chemical modification of the silica surface occurring during synthesis that “seals off” any exposed silanol sites of the silica matrix preventing basic ions from dissolving the silica matrix (silicon-oxygen bonds). Therefore, silica-based Diphonix resin is expected to be more effective at high pH (>11) than other silica-based ion exchangers.

Organic Tolerance Many of the waste streams generated within the DOE combine actinides, other radionuclides and organic contaminants under highly acidic or highly basic conditions. This combination of contaminants eliminates the effectiveness of most ion exchangers and significantly restricts treatment options. Polystyrene-based Diphonix resin, like all ion exchange resins with organic matrices, is subject to organic fouling which occurs when organic contaminants form a coating over the polystyrene-based resin bead. A silica-based Diphonix resin is less likely to foul because the inorganic silica-based matrix will not be as easily coated with an organic contaminant. Because it resists fouling, it is likely to be more effective in treating the organic-containing wastes of the DOE.

FUTURE ACTIVITIES

The objective is to synthesize an improved version of Diphonix resin that is more broadly applicable to the problems at the DOE complex. The project's goal is to retain the advantages of DMBP resins, namely rapid kinetics, metal ion selectivity and acid-stability, while addressing specific challenges that arise within the DOE complex, including requirements for radiolytic stability, constraints on the disposal of the ultimate waste, requirements for high metal ion loading at both acidic and basic pH, and organic contamination of the waste streams. The approach is to extend the diphosphonic acid functionality that has been successfully commercialized for actinide and heavy metal removal from a polystyrene-based matrix to a silica-based matrix. The performance of the diphosphonic acid functionality (polystyrene-based Diphonix resin) is well understood and

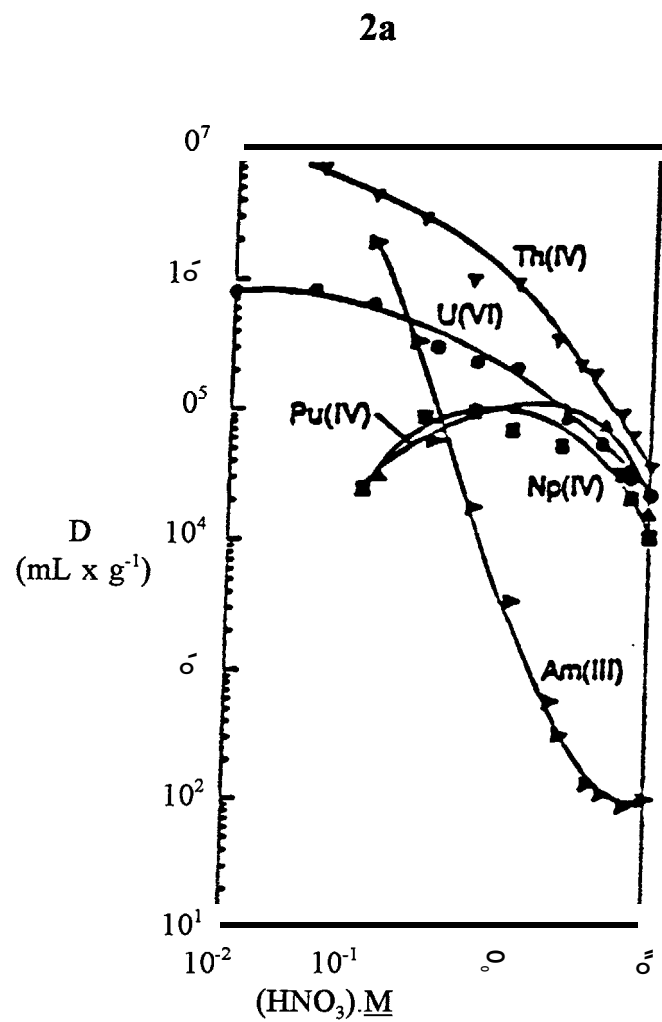
documented and preliminary laboratory synthesis studies indicate that the diphosphonic functionality can be added to a silica-based matrix. The project significantly builds on the previous work, allowing a detailed assessment of technical and financial feasibility of producing a silica-based Diphonix resin that performs comparably to polystyrene-based Diphonix resin.

The technical risks associated with the project have been mitigated by developing a coherent and comprehensive project work plan based on the results of preliminary silica-based Diphonix resin synthesis studies and the extensive data generated during performance testing of polystyrene-based Diphonix resin. Moreover, the go/no go decisions points, two of which include economic evaluations, are judiciously positioned at four critical junctures in the project and further mitigate the technical and financial risks of developing a new resin product. It is expected that the proposed resin will provide an economic treatment method for a broad range of DOE actinide-contaminated waste streams. Eichrom's experience in the scale-up and commercialization of the polystyrene-based Diphonix resin will enable an effective comparative evaluation of the economic potential of the proposed silica-based resin.

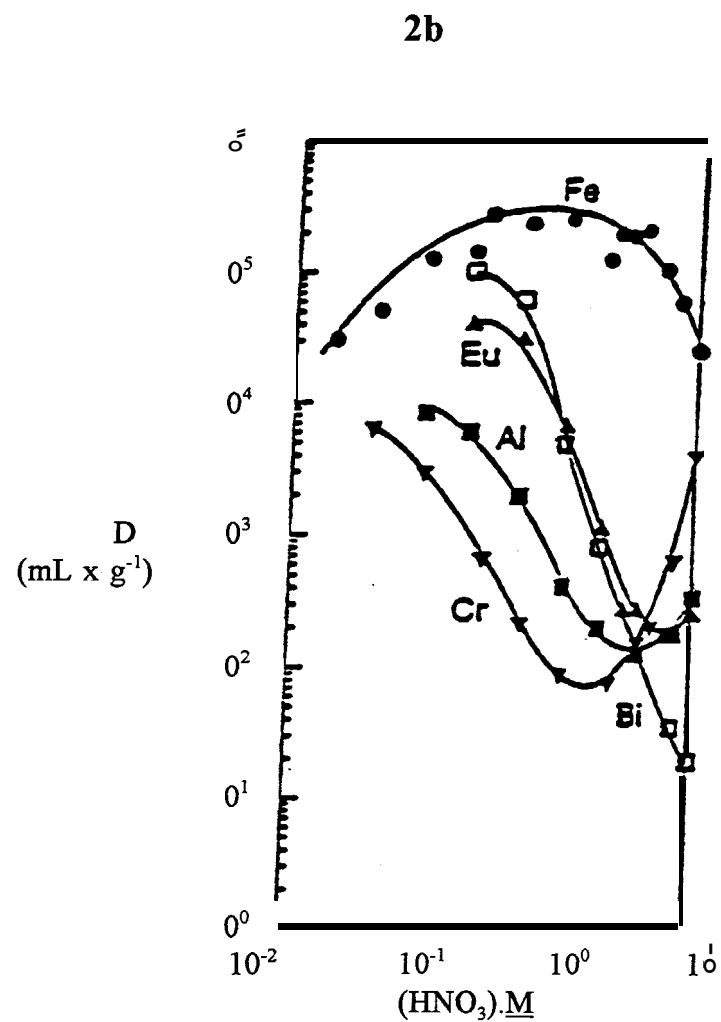
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Figure 2



Nitric Acid Dependencies of Actinide Ion Uptake by Diphonix Resin



Nitric Acid Dependencies of Trivalent Ion Uptake by Diphonix Resin